
(12) **UK Patent Application** (19) **GB** (11) **2 173 781 A**

(43) Application published 22 Oct 1986

(21) Application No 8608889

(22) Date of filing 11 Apr 1986

(30) Priority data

(31) 8501773 (32) 11 Apr 1985 (33) SE

(71) Applicant
Berol Kemi AB (Sweden),
Box 851, S-4444 01 Stenungsund, Sweden

(72) inventors
Axel Ingemar Thebrin,
Pia Christina Thebrin

(74) Agent and/or Address for Service
J. A. Kemp & Co., 14 South Square, Gray's Inn,
London WC1R 5EU

(51) INT CL⁴
C01F 11/18 11/46

(52) Domestic classification (Edition H):
C1A 114 328 329 D37 D45 D8 G47 G47D45 G47D8
PG1A
U1S 1397 2249 C1A

(56) Documents cited
US 3645677

(58) Field of search
C1A
Selected US specifications from IPC sub-class C01F

(54) **Water slurry of gypsum or chalk**

(57) A slurry of gypsum or chalk in water containing a dispersing agent in the form of a surface active organic sulphate or sulphonate, and a complexing agent in the form of an inorganic phosphate or an organic amine. The slurry has low viscosity at the desired content of gypsum or chalk.

GB 2 173 781 A

SPECIFICATION

Water slurry of gypsum or chalk

- 5 The present invention relates to a water slurry of gypsum or chalk containing an anionic surface active agent as a dispersing agent, as well as an inorganic phosphate or organic amine compound as a complexing agent. 5
- Aqueous slurries of fine-particulate gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, or chalk, CaCO_3 , have been used as paper coating agents, within the building industry and as fillers for many applications. In order to obtain a homogeneous dispersion, carboxymethyl cellulose (CMC) is usually added as a dispersing agent. However, 10
- 10 water slurries based on gypsum or chalk have found limited use because it is difficult to produce a slurry having a sufficiently high gypsum or chalk content and, at the same time, low viscosity and high stability. Normally, a dry content of about 65–80% by weight is desired in such slurries. 10
- It has now proved to be possible to reduce the viscosity of an aqueous slurry of gypsum or chalk at a given dry content of usually about 50% and, in some useful embodiments, of 70% or more, as compared to the 15
- 15 viscosities obtained with a corresponding slurry but with CMC as a dispersing agent. According to the invention, the slurry contains, besides fine-particulate gypsum or chalk and water, a dispersing agent in the form of a surface active organic sulphonate or sulphate in an amount of 0.2–3, preferably 0.4–1.5% by weight, based on the weight of the composition, and a complexing agent in the form of an inorganic phosphate or an 20
- 20 organic amine compound in an amount of 0.1–3, preferably 0.2–1.5% by weight, based upon the weight of the composition. The reason for this surprising viscosity reduction is not known, but one theory is that divalent cations occurring in the slurry serve as a coupling member between anionic sites on the gypsum or chalk particles and the anionic compound. In this manner, a bimolecular layer is formed, whereby an electrostatic dispersion of the particles is made possible. A slurry of gypsum or chalk always contains a 25
- 25 certain amount of Ca^{2+} ions because gypsum and chalk is sparingly water-soluble. Due to the ability of the complexing agent to form complex with Ca^{2+} it is believed according to this theory that the Ca^{2+} function as a coupling member is facilitated. In many cases, the amount of Ca^{2+} is insufficient in relation to the total amount of cations in the aqueous phase, for which reason in such cases further bi- or polyvalent cations should be introduced into the slurry. Preferably, this is done by adding the anionic compound in the form of 30
- 30 a divalent salt, preferably Ca^{2+} . Suitable anionic surface active sulphonates and sulphates according to the invention are, for example, alpha-olefin sulphonate derived from alpha-olefins having 8–22 carbon atoms, alkyl sulphate having 8–22, preferably 12–18 carbon atoms, and alkyl benzene sulphonate having 10–30, preferably 12–24 carbon atoms. They may be present in the form of a salt and/or as an acid, salts with divalent cations, such as calcium and magnesium, being preferred. 30
- Examples of suitable inorganic complexing phosphates according to the invention are conventional 35
- 35 complexing phosphates, such as tetrasodium pyrodiphosphate, sodium hexametaphosphate, trisodium phosphate, sodium hydrogen pyrophosphate, sodium tripolyphosphate, and corresponding potassium compounds. Of these compounds tetrasodium pyrophosphate, sodium hexametaphosphate, and trisodium phosphate are preferred.
- The complex forming organic amine compounds comprise any conventional complexing agent of this 40
- 40 group and especially those amine compounds having at least one primary or secondary nitrogen and at least one hydroxyl group, such as monoethanolamine, diethanolamine, hydroxypiperazine, and aminoethyl ethanolamine. Hydroxypiperazine and aminoethyl ethanolamine are preferred.
- Gypsum in finely divided form is obtained in large quantities in the production of phosphoric acid from the phosphate-containing minerals via recrystallisation. Chalk suitable in the preparation is precipitated chalk 45
- 45 but chalk of other qualities may also be used. The final selection of qualities of gypsum and chalk is of course ruled by the intended use of the slurry. The particle size and the particle size distribution affect materially the viscosity of the slurry. The average particle size suitably is below 100 μm , preferably below 25 μm . A particle size distribution having a high proportion of fines facilitates close packing of the particles and also facilitates the preparation of slurries having high filler contents at suitable viscosities. A suitable milling technique to 50
- 50 obtain the desired particle size distribution is described in international patent application No. 83/04046. Finely divided material may also be achieved by milling the aqueous slurry itself. The amount of gypsum and/or chalk in the slurry is normally about 65–85% by weight.
- In addition to the said surface active anionic compounds and complexing agents, the slurry may contain conventional additives, such as bactericides, defoaming agents, pH-controlling agents, and stabilizers. In 55
- 55 some cases, the stabilisation obtained with the dispersing compound alone may be further improved by the addition of supplementing stabilisers. Such stabilisers are primarily clays, such as bentonite coated with a hydrophobing agent. Other useful stabilisers are cellulose ethers, such as CMC, methyl cellulose and ethyl hydroxy cellulose. The above-mentioned cellulose ethers also have a useful water-retaining ability. The amount of stabilisers preferably is 0.02–0.5, preferably 0.05–0.3% of the weight of the slurry. 60
- 60 The slurry of the invention may also besides gypsum and chalk contain additional particulate material, such as talc and clays. Such additional particulate material can be added when formulating a slurry of the invention. This method is suitable in the case the amount of additional particulate material is low. Another method is to prepare a particular aqueous slurry containing the additional particulate material and mix this slurry with a slurry of the invention.
- 65 The invention will be further illustrated by the following Example. 65

Example

To 100 parts of finely divided gypsum (runs 1-19 and references A-D) or chalk (runs 20 and 21 and references E and F) having a water content of 25% by weight and a particle size of 1-25 μ m, an anionic surface active compound and a complexing agent in the form of an inorganic phosphate or an organic amine compound as well as sodium hydroxide and additional water were added, such that pH 9 and the dry contents stated in the Table below were obtained. The mixture was rapidly agitated so that a dispersion was obtained. The viscosity of the dispersion was determined by means of a Brookfield RLV at 30 and 60 rpm. Stability was determined visually after 24 hours according to a scale of 1-2, 1 meaning that no phase separation has occurred and 2 that two distinct phases could be observed. The following results were obtained.

TABLE

Run	Anionic surface active compound	g/kg particulate material	Complexing agent	g/kg particulate material	Dry content % by weight	Viscosity 30 rpm	Viscosity 60 rpm	Stability
1	Ca-dodecylbenzene sulphonate	10	Na ₄ -pyrophosphate	5	67.0	240	160	1
2	"	10	"	3	69.7	300	140	1
3	"	10	"	2	71.8	420	390	1
4	"	10	"	1	72.0	560	560	1
5	"	7	Na ₆ -metaphosphate	6	73.4	350	300	1
6	"	7	Na ₄ -pyrophosphate	6	73.5	800	750	1
7	"	11	Na ₆ -metaphosphate	2	72.8	920	800	1
8	"	11	Na ₄ -pyrophosphate	2	75.0	580	400	1
9	"	7	Na ₆ -metaphosphate	3	75.0	280	250	1
10	"	11	K ₄ -pyrophosphate	2	72.1	590	410	1
11	Dodecylbenzene sulphononic acid	5	Na ₄ -pyrophosphate	1	70.9	1240	900	1
12	"	5	"	1	72.0	830	790	1
13	Ca-C ₁₄₋₁₈ olefin sulphonate	10	"	3	70.2	380	240	1
14	Ca-C ₁₄₋₁₈ -alkyl sulphate	10	"	3	70.1	380	260	1
15	Dodecylbenzene sulphononic acid	5	Monoethanolamine	5	72.1	600	450	1
16	"	5	Diethanolamine	5	70.1	1000	800	1
17	"	5	Hydroxyethyl piperazine	5	70.2	100	100	1
18	"	5	Aminoethyl ethanolamine	5	70.1	50	50	1
19	"	4	"	4	70.0	400	300	1
20	"	3	"	2	73.2	350	250	1
21	"	3	Na ₆ -metaphosphate	2	73.0	390	300	1
Reference A			Additive					
B			Carboxymethyl cellulose	15	72.0	1300	1500	2
C			Ca-calcium bensene	15	72.1	3100	1800	2
D			Na ₄ -pyrophosphate	15	72.0	5000	5000	2
E			Na ₆ -metaphosphate	15	72.0	5000	5000	2
F			"	3	73.0	2000	2000	2
			Aminoethyl ethanolamine	3	73.0	2100	2000	2

CLAIMS

1. A water slurry of gypsum or chalk comprising as dispersing agent 0.2 to 3% by weight of a surface active anionic organic sulphonate or sulphate and, as complexing agent, 0.1 to 3% by weight of an inorganic phosphate or organic amine compound.
2. A slurry according to claim 1 comprising 0.4 to 1.5% by weight of the surface active anionic organic compound.
3. A slurry according to claim 1 or claim 2 comprising 0.2 to 1.5% by weight of the inorganic phosphate or

- organic amine compound.
4. A slurry according to any one of claims 1 to 3 wherein the surface active anionic organic compound is alkyl benzene sulphonate or the calcium salt thereof.
5. A slurry according to any one of claims 1 to 4 wherein the complexing agent is tetrasodium pyrophosphate, sodium hexametaphosphate or trisodium phosphate. 5
6. A slurry according to any of claims 1 to 5 wherein the complexing agent is an organic amine compound having at least one primary or secondary nitrogen and at least one hydroxyl group.
7. A slurry according to claim 6 wherein the organic amine compound is aminoethyl ethanolamine or hydroxyethyl piperazine.
- 10 8. A slurry according to any one of claims 1 to 7 comprising, as stabiliser, a clay or a cellulose ether in an amount of 0.02 to 0.5% by weight. 10
9. A slurry according to any one of claims 1 to 8 wherein the average particle size is below 100µm.
10. A slurry according to claim 9 wherein the average particle size is below 25µm.
11. A slurry according to any one of claims 1 to 10 comprising from 65 to 85% total weight of gypsum and 15 chalk. 15
12. A slurry according to claim 1 and substantially as hereinbefore described with reference to the Example.
13. A process for producing a slurry according to any one of claims 1 to 12 comprising bringing into association water, gypsum or chalk, a surface active anionic organic sulphonate or sulphate and an inorganic phosphate or organic amine compound. 20
14. A process according to claim 13 and substantially as hereinbefore described with reference to the Example.
15. A process for producing coated paper comprising applying a slurry according to any of claims 1 to 12 to paper.
- 25 16. Use of a slurry according to any one of claims 1 to 12 in the coating of paper. 25